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IS 1035 (1972): Methods of sampling and test for bleaching
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“Knowledge is such a treasure which cannot be stolen”

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REAFFIRMED

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Indian Standard
**METHODS OF SAMPLING AND TEST FOR
BLEACHING EARTHS
(*Second Revision*)**

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**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

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Indian Standard

METHODS OF SAMPLING AND TEST FOR BLEACHING EARTHS

(*Second Revision*)

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Indian Standard
**METHODS OF SAMPLING AND TEST FOR
BLEACHING EARTHS**
(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 24 March 1972, after the draft finalized by the Oils and Oilseeds Sectional Committee had been approved by the Chemical Division Council and the Agricultural and Food Products Division Council.

0.2 Large quantities of different types of bleaching earths are used in the country for bleaching vegetable oils. With a view to introducing uniform methods of sampling and test for these materials in the country, this standard was first published in 1957.

0.3 In this revision, the method of test for decolorizing power is being substituted by that for bleaching efficiency. Also provision has been made for the use of two alternative oils for the test depending on the application.

0.4 For particle size, the use of IS Sieves conforming to IS : 460-1962* is prescribed. Where IS Sieves are not available, other standard sieves as judged from aperture size may be used.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard prescribes the methods of sampling and test for bleaching earths. It covers the methods for determining moisture, bulk density, sieve analysis, acidity or alkalinity, bleaching efficiency, filtrability, oil retention and ash.

*Specification for test sieves (*revised*).

†Rules for rounding off numerical values (*revised*).

2. SAMPLING

2.1 General Precautions — In drawing, preparing, storing and handling samples, the following precautions shall be observed:

- a) Samples shall not be taken in a place exposed to weather.
- b) The sampling instruments shall be clean and dry when used.
- c) Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.
- d) The samples shall be placed in clean, dry, air-tight glass or other suitable containers on which the material has no action.
- e) The sample containers shall be of such a size that they are almost, but not completely, filled with the sample.
- f) Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, the year of manufacture and other important particulars of the consignment.
- g) Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

2.2 Scale of Sampling

2.2.1 Lot — All the containers, in a single consignment, of the same size, type, grade of material and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers of different sizes, types, grades or batches of manufacture, the containers belonging to the same size, type, grade and batch of manufacture shall be grouped together and each such group shall form a separate lot.

2.2.1.1 Samples shall be tested from each lot separately for ascertaining conformity of the material to the requirements of any specification.

2.2.2 The number of containers to be selected from a lot shall depend upon the size of the lot and shall be in accordance with Table 1.

2.2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables may be used. In case such tables are not available, the following procedure may be adopted.

2.2.3.1 Starting from any container count them as 1, 2, 3,....., up to r and so on in one order. Every r th container thus counted shall be chosen to give sample for test, r being the integral part of N/n , where N is the total number of containers in the lot and n the number of containers to be selected (see Table 1).

TABLE 1 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING*(Clauses 2.2.2 and 2.2.3.1)*

Lot Size (<i>N</i>)	SAMPLE SIZE (<i>n</i>)
(1)	(2)
3 to 50	3
51 „ 200	4
201 „ 400	5
401 „ 650	6
651 and above	7

2.3 Test Samples and Referee Samples

2.3.1 To ensure that the sample taken from each container is fairly representative, the material shall be drawn from different parts of the container with the help of a suitable sampling implement so as to give a representative sample for the container. The approximate quantity of material to be drawn from a container shall be nearly equal to thrice the quantity required for testing purposes as indicated in **2.4**.

2.3.2 Out of each of these portions, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample sufficient for carrying out triplicate determinations for all the characteristics specified under **2.4.2**. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

2.3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be marked for the purchaser, another for the supplier and the third for the referee.

2.3.4 All the individual and composite samples shall be transferred to separate containers. Each container shall then be sealed air-tight with stoppers and labelled with full identification particulars [see **2.1 (f)**].

2.3.5 The referee test sample consisting of a composite sample and a set of *n* individual samples shall bear the seals of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

2.4 Number of Tests

2.4.1 Tests for the determination of moisture and bleaching efficiency as specified in the relevant material specifications, shall be conducted on each of the individual samples separately.

2.4.2 Tests for the determination of the remaining characteristics in the material specification shall be conducted on the composite sample.

2.5 Criterion for Conformity

2.5.1 A lot shall be considered as conforming to this specification if it satisfies the criteria as given in **2.5.2.1** and **2.5.3**.

2.5.2 The test results for the determinations of moisture, and bleaching efficiency shall be recorded as shown in Table 2. The mean and the range shall be calculated as follows and shall be recorded in col 3 and 4 respectively of Table 2:

$$\text{Mean } (\bar{X}) = \frac{\text{the sum of the test results}}{\text{number of test results}}$$

$$\text{Range } (R) = \text{the difference between the maximum and the minimum values of the test results.}$$

2.5.2.1 The corrected mean as shown in col 5 of Table 2 shall be calculated. The lot shall be considered to have satisfied the requirement for a characteristic if the condition given in col 6 of Table 2 is satisfied.

TABLE 2 CRITERIA FOR CONFORMITY

(Clauses 2.5.2 and 2.5.2.1)

SL No.	CHARACTERISTIC	TEST RESULTS		CORRECTED MEAN	CRITERION FOR CONFORMITY
		Mean	Range		
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture	\bar{X}_1	R_1	$\bar{X}_1 + 0.6R_1$	Corrected mean \leq specified value
ii)	Bleaching efficiency	\bar{X}_2	R_2	$\bar{X}_2 - 0.6R_2$	Corrected mean \geq specified value

2.5.3 The composite sample when tested for the remaining characteristics not tested in **2.5.2** shall satisfy the corresponding requirements for them.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (IS : 1070-1960*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water, distilled quality (revised).

4. MOISTURE CONTENT

4.0 Principle of the Method — A known mass of the material is kept at 105 to 110°C till no further loss in mass is recorded. Difference in mass gives moisture content.

4.1 Procedure — Weigh accurately about 10 g of the material in a tared, flat-bottom crystallizing dish with a cover. Place the uncovered dish in an oven maintained at 105 to 110°C for 3 hours. Cover the dish, cool in a desiccator, and weigh. Repeat the above procedure of heating for 30 minutes at 105 to 110°C, cooling and weighing until the difference between two successive weighings does not exceed 5 mg.

4.2 Calculation

$$\text{Moisture content, percent by mass} = \frac{100 (M - m)}{M}$$

where

M = mass in g of the material taken for the test, and

m = mass in g of the material after drying.

5. BULK DENSITY

5.0 Principle of the Method — A known mass of the material is taken in a measuring cylinder avoiding compression. The cylinder is made to strike against a rubber pad under specified conditions and bulk density known by the ratio of the mass of the material and the volume it occupied finally.

5.1 Apparatus — The apparatus (*see* Fig. 1) shall satisfy the following requirements.

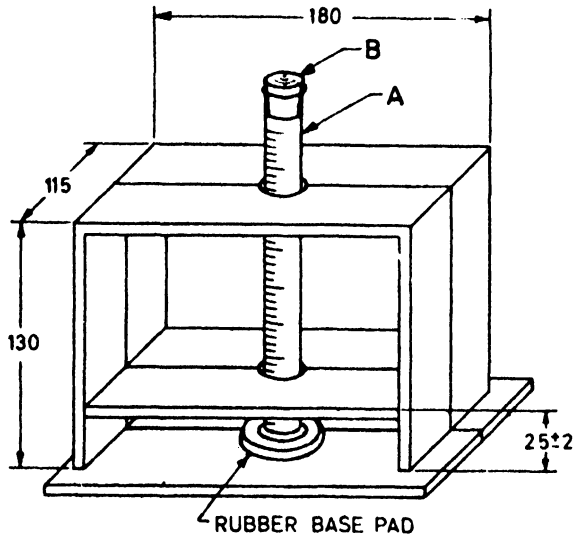
5.1.1 The base of the graduated measuring cylinder A shall be ground flat. The empty measuring cylinder A , together with the rubber bung B , shall weigh 250 ± 5 g.

5.1.2 The measuring cylinder A shall be graduated in 2-ml subdivisions over the range of 25 to 250 ml.

5.1.3 The distance between zero and 250-ml graduation on the measuring cylinder A shall be not less than 220 mm and not more than 240 mm.

5.1.4 The distance between the flat-ground part of the base of the measuring cylinder A and the upper base pad, when the measuring cylinder A is raised to the full height, shall be 25 ± 2 mm.

5.1.5 The rubber base pad shall have Shore Hardness Number of 25 to 50.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF BULK DENSITY

5.2 Procedure — Take 80 to 100 g of the material. Hold the tared measuring cylinder in a slightly inclined position and slide the material into it up to the 100-ml mark, taking care to see that the material is not compressed. Weigh the measuring cylinder with the contents. Stopper the measuring cylinder. Raise it to a height of 25 ± 2 mm (see Fig. 1) and then release it to strike against the rubber pad. Repeat this process 50 times and read the exact volume occupied by the packed material.

5.3 Calculation

$$\text{Bulk density, in g per ml} = \frac{M}{V}$$

where

M = mass in g of the material taken for the test, and

V = final volume in ml of the material.

6. SIEVE ANALYSIS

6.0 Principle of the Method — The material is sieved through standard sieves and percent of the material passing through various sieves calculated.

6.1 Apparatus

6.1.1 Camel Hair Brush

6.1.2 Rubber Cushion or Thick Piece of Flat Rubber

6.1.3 Test Sieves — IS Sieves 125-micron, 63-micron and 53-micron conforming to IS : 460-1962*.

6.1.3.1 Mechanical shaker — Wherever possible a mechanical shaker may be used.

6.2 Procedure — Clean and thoroughly dry the sieves before use. Nest the sieves in order with 125-micron IS Sieve on top. Weigh accurately 100 g of the well-mixed material and transfer it into the top sieve. Shake the sieves simultaneously and occasionally tap the bottom sieve on the rubber cushion. When the portion of the material passing through the bottom sieve appears to be negligible (less than 0.2 g per minute of shaking), remove the top sieve and shake this sieve separately over a clean, white glazed paper to ensure that no more of the material passes through. Using the camel hair brush, transfer from the paper any material that has passed through the 125-micron IS Sieve to 63-micron IS Sieve. Repeat the same procedure with 63-micron IS Sieve and 53-micron IS Sieve. Transfer the residue remaining on each sieve to convenient tared containers with the aid of a camel hair brush, and weigh each separately.

6.3 Calculation

$$\left. \begin{array}{l} \text{Material retained on or passing} \\ \text{through any given sieve, per-} \\ \text{cent by mass} \end{array} \right\} = \left\{ \begin{array}{l} \text{Mass in g of the material re-} \\ \text{tained on or passing through} \\ \text{the given sieve} \end{array} \right.$$

6.3.1 The sum of the percentages of the material retained on each sieve plus the percentage passing through 53-micron IS Sieve shall be 100.

7. ACIDITY OR ALKALINITY

7.0 Principle of the Method — The material is digested with distilled water. Bromothymol blue indicator is added to the filtered solution. Depending on the acidity or alkalinity, as indicated by the indicator, the solution is titrated using solution of alkali or acid of known strength.

7.1 Reagents

7.1.1 Bromothymol Blue Indicator Solution — 0.1 percent (*m/v*) in 50 percent rectified spirit (see IS : 323-1959†).

*Specification for test sieves (*revised*).

†Specification for rectified spirit (*revised*).

7.1.2 Standard Sodium Hydroxide Solution — approximately 0.02 N.

7.1.3 Standard Sulphuric Acid Solution — approximately 0.02 N.

7.2 Procedure — Weigh accurately about 2 g of the material and transfer it to a 400-ml beaker. Add 200 ml of water, mix well with a glass rod and heat to boiling. Digest for 10 minutes and allow to stand. Filter, cool the filtrate and make up the volume to 250-ml mark in a volumetric flask. Take about 5 ml of this solution in a test-tube and add a few drops of bromothymol blue indicator solution. If the solution is coloured yellow, determine its acidity (*see* 7.2.1) and, if it is blue, determine its alkalinity (*see* 7.2.2).

NOTE — Before carrying out the test it should be ascertained that the glass of the beaker as well as the rod to be used for boiling does not impart any alkalinity to water. This can be done by boiling only distilled water in 400-ml beaker and testing it for alkalinity. The beaker and glass rod shall be rinsed with distilled water before performing the test.

7.2.1 Determination of Acidity — To a 100-ml aliquot of the solution (*see* 7.2) add a few drops of bromothymol blue indicator solution and titrate with standard sodium hydroxide solution until the colour of the solution changes to greenish blue.

7.2.1.1 Calculation

$$\text{Acidity (expressed as H}_2\text{SO}_4\text{), percent by mass (on dry basis)} = \frac{1\,225\,VN}{M_1(100 - M)}$$

where

V = volume in ml of standard sodium hydroxide solution used for titration,

N = normality of standard sodium hydroxide solution,

M_1 = mass in g of the material taken for the test, and

M = moisture content in percent by mass of the material taken for the test (*see* 4).

7.2.2 Determination of Alkalinity — To a 100-ml aliquot of the solution (*see* 7.2), add a few drops of bromothymol blue indicator solution and titrate with standard sulphuric acid solution until the greenish blue colour of the solution changes to almost yellow.

7.2.2.1 Calculation

$$\text{Alkalinity (expressed as NaOH), percent by mass (on dry basis)} = \frac{1\,000\,VN}{M_1(100 - M)}$$

where

V = volume in ml of standard sulphuric acid solution used for titration,

N = normality of standard sulphuric acid solution,

M_1 = mass in g of the material taken for the test, and

M = moisture content in percent by mass of the material taken for the test (see 4).

8. BLEACHING EFFICIENCY

8.0 Principle of the Method — Depending on the intended use of the bleaching earth, either alkali refined groundnut oil or washed cottonseed oil is taken for the test. The oil selected is treated with the material under test, and the colour of the oil, both before and after the test, is measured in Lovibond units. From the colours of the original and the bleached oil, the decolorizing power of the material is calculated. The bleaching efficiency is obtained by comparing this figure with that of standard bleaching earth on the same oil under the same conditions of test.

8.1 Reagents

8.1.1 Alkali Refined Groundnut Oil — of colour 10 ± 2 units when expressed as $Y + 5R$ on the Lovibond scale in a 1-in cell;

OR

Washed Cottonseed Oil — of colour 25 ± 2 units, when expressed as $Y + 10R$ on the Lovibond scale in a $\frac{1}{2}$ -in cell.

8.1.2 Carbon Dioxide Gas — from a cylinder. It should be dried by passing it through a drying bottle.

8.2 Apparatus

8.2.1 Drying Bottles — two, capacity 500 ml; one containing concentrated sulphuric acid.

8.2.2 Glass Cells — 25 mm (or 1 in) and $\frac{1}{2}$ -in, made of clear, colourless glass.

8.2.3 Lovibond Tintometer

8.2.4 Round Bottom Flask — three-neck, 500 ml capacity (see Fig. 2).

8.2.5 Stirrer — made of glass with one end bent in the form of a triangle or paddle (see Fig. 2). The upper end should be connected to a pulley which is driven by a small electric motor.

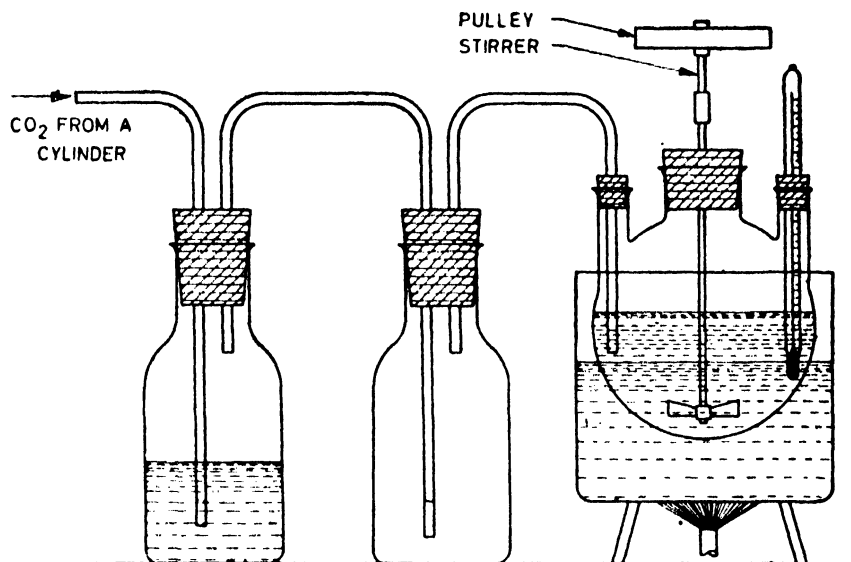


FIG. 2 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF DECOLORIZING POWER

8.3 Procedure — Weigh accurately 200.0 ± 0.1 g of the alkali refined groundnut oil or washed cottonseed oil (see 8.1.1) in the round-bottom flask. Immerse the flask in a water-bath maintained at 90 to 95°C and slowly bubble carbon dioxide gas through the oil. When the temperature of the oil has reached 90°C, momentarily remove the cork and introduce 2.0 g of the bleaching earth. Start the stirrer and adjust its speed to about 350 rpm. The bottom end of the stirrer should be adjusted to a level at which intimate mixing is ensured. Maintain the temperature at 90°C for 20 minutes, and then stop stirring. Remove the flask from the water-bath, allow the oil to cool in the atmosphere of carbon dioxide and filter the oil through filter paper (Whatman No. 1 or equivalent). Determine the colour readings of the original oil and the bleached oil in the glass cell in a Lovibond tintometer.

8.3.1 Repeat the test described in 8.3 using standard bleaching earth in place of the sample of the bleaching earth under test; and determine the colour readings of the original and bleached oil, using another portion of the same oil as that used for the test and in the same size of the cell.

8.4 Calculation

a) Decolorizing power of the material under test, D_4 , percent

$$= \frac{100 (A - B)}{A}$$

where

A = colour units of the original oil, expressed either as $Y + 5R$, or as $Y + 10R$, depending on the oil used (*see 8.1.1*), on the Lovibond scale; and

B = colour units of the bleached oil, expressed as above.

b) Bleaching efficiency, percent = $\frac{D_t}{D_s} \times 100$

where

D_s = decolorizing power, percent, of the standard bleaching earth (*see 8.3.1*) on the same sample of the oil and using the same Lovibond cell.

9. FILTRABILITY AND OIL RETENTION

9.0 Principle of the Method

- a) *Filtrability*— is determined by noting the time taken by oil in filtering through a bed of the material, prepared under specified conditions.
- b) *Oil Retention* — The oil retained by the cake of the material formed during filtration, is extracted with solvent under specified conditions and oil retention determined.

9.1 Reagents

9.1.1 Alkali Refined Groundnut Oil

9.1.2 *Solvent* — petroleum ether (conforming to solvent Grade 60/80 of IS : 1745-1966*).

9.2 Apparatus

9.2.1 *Buchner Funnel* — 9 cm inside diameter immersed in hot water-bath.

9.2.2 *Filter Flask* — one litre capacity.

9.2.3 *Filter Pump or Vacuum Pump*

9.2.4 *Soxhlet Extraction Apparatus*

9.2.5 *Stop Watch*

*Specification for petroleum hydrocarbon solvents (*first revision*).

9.3 Procedure

9.3.1 Determination of Filtrability

9.3.1.1 Weigh 200 ± 0.1 g of the refined groundnut oil in a 500-ml beaker. Weigh accurately 10 g of the material and add to the oil. Heat the oil to 90°C mixing thoroughly with a glass rod. Wet the filter paper (Whatman No. 1 is suitable) placed inside the Buchner funnel with a little groundnut oil, taking care to avoid channeling effect. Heat the water-bath and maintain its temperature at 90°C . Start the vacuum pump. Mix the contents of the beaker thoroughly and immediately pour the entire mixture in the Buchner funnel. Adjust vacuum to absolute pressure of 100 ± 10 mm of mercury. In another beaker keep ready 200 g of groundnut oil (without bleaching material) heated up to 90°C (oil A). As soon as the film of oil on the cake in the Buchner funnel disappears, pour into the Buchner funnel over the bed of bleaching material second batch of groundnut oil heated to 90°C (oil A). Start the stop watch as soon as the filtered oil starts flowing from the funnel. Maintain the vacuum at the absolute pressure of 100 ± 10 mm of mercury. Note the time when the oil film on the cake in the Buchner funnel disappears. At this point the vacuum will also drop.

9.3.1.2 Filtrability is the time in minutes required for the second batch of oil (oil A) to filter through the cake of bleaching material.

NOTE — Continue filtration till the rate of filtration falls to a drop per 15 seconds. Stop the vacuum pump and remove the cake from the Buchner funnel for the determination of oil retention as described in 9.3.2.

9.3.2 Determination of Oil Retention — Weigh accurately by difference about 5 g of the cake on to a filter paper (Whatman No. 42 is suitable), roll and enclose it in a second filter paper folded in such a manner as to prevent the cake from escaping (the second filter paper is left open at the top to serve as a thimble). Place a piece of absorbent cotton in the top of the thimble to distribute the solvent evenly over the material. Place the wrapped material in the Soxhlet extraction apparatus. Put approximately 75 ml of the solvent into the tared extraction flask before attaching to the tube. Heat on a water-bath or electric hot plate at such a rate that the solvent drops from the condenser on the centre of the thimble at the rate of at least 150 drops per minute. Keep the volume of the solvent fairly constant by adding enough to make up the loss due to evaporation. Continue extraction for 3 hours. Cool and disconnect the extraction flask. Evaporate the solvent on a steam- or water-bath until no odour of it remains. A gentle stream of clean, dry air may be used to facilitate removal of the solvent. Cool to room temperature, carefully remove any moisture or dirt from the outside of the flask and weigh. Repeat heating, cooling and weighing until the difference between two successive weighings does not exceed 1 mg. Find the mass of the oil retained in the extraction flask.

9.3.2.1 Calculation

$$\begin{array}{l} \text{Oil retained by the material,} \\ \text{percent by mass} \end{array} = \frac{100 \, m}{M}$$

where

m = mass in g of the oil retained in the extraction flask, and

M = mass in g of the filter cake for the determination of oil retention.

NOTE — The determination of oil retention must be done soon after the determination of filtrability (see 9.3.1).

10. ASH

10.0 Principle of the Method — The material is heated at 850 to 900°C and percentage of the ash determined.

10.1 Apparatus

10.1.1 Muffle Furnace — Electrically heated, with suitable ventilation and temperature control device (850 to 900°C).

10.2 Procedure — Render the sample homogeneous by kneading in a mortar. Weigh accurately about 3 g of the material in a tared porcelain dish. Ignite in a muffle furnace, and raise the temperature in 2½ hours to 850 to 900°C. Maintain at that temperature for about 3 hours, then cool the dish in a desiccator and weigh. Repeat the procedure of heating, cooling and weighing at ½ hour intervals until the difference between two successive weighings does not exceed 1 mg.

10.3 Calculation

$$\text{Ash, percent by mass} = \frac{100 \, M_1}{M_2}$$

where

M_1 = mass in g of the material after ashing, and

M_2 = mass in g of the material taken for the test.

11. PERFORMANCE TEST

11.1 As confirmatory tests on bleaching efficiency (see 8), and filtrability and oil retention (see 9), the purchaser may carry out performance tests in the plant on these characteristics subject to agreement between the purchaser and the supplier.

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